

Syntactic foams consolidated with starch: mixing behaviour for post-mould processing

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Abstract. The mixing required for post-mould gelatinisation of syntactic foams using starch as binder was studied. It was found that starch particles tend to adhere to hollow microspheres during mixing, forming agglomerates. A transition in the buoyancy of microsphere-starch phases was found at a particular volume fraction of starch. The transitional point was close to a volume fraction of starch at which a calculated relative density for a system consisting of multiple starch particles per microsphere was unity. Starch-microsphere inter-distance appears to be an important parameter affecting starch content in an agglomeration.

Introduction

Syntactic foams are particulate composites made of pre-formed hollow microspheres and binder [1]. A wide range of different types of syntactic foams can be made by selecting different materials and consolidating techniques. As previously discussed in reference [2], the buoyancy method has recently been used with a wide range of binder contents and at low costs, therefore widening applicability of syntactic foams. Also it allows us to use starch as binder for manufacturing potential building materials such as interior wall boards and ceiling panels. The advantages of starch over other binders such as epoxies and phenolics are that it is readily available, environmentally friendly, inexpensive and renewable.

A main purpose of the present work was to investigate mixing behaviour of starch particles and ceramic hollow microspheres in ‘post-mould processing’ [2, 3] of syntactic foams.

Materials and Method

Hollow Microspheres. Ceramic hollow microspheres supplied by Envirospheres Pty Ltd, Australia were used. Two different size groups (or commercial grades), SL75 and SL300, were employed.

Microsphere and starch particle sizes were measured using a Malvern 2600C laser particle size analyser and mean sizes were found to be 53, 178, and 47 μm for SL75, SL300, and starch respectively. They were found to be of approximately Gaussian distribution as previously shown in reference [2]. Particle densities and bulk densities of the two hollow microsphere groups were also measured using a Beckman Air Comparison Pycnometer (Model 930) and a measuring cylinder (capacity 250 cc) respectively. Three hundred taps were conducted for each bulk density measurement. An average of five measurements was taken for each size group and is available in reference [2].

Starch Binder. Potato starch (Tung Chun Soy & Canning Company, Hong Kong) was used as binder for hollow microspheres. Particle density of the potato starch was measured using a Beckman Air Comparison Pycnometer (Model 930) and an average of three measurements was

found to be 1.50 g/cc. Bulk density was also measured using a measuring cylinder with a tapping device (300 taps were conducted) and an average of five measurements was found to be 0.85 g/cc. SEM images of starch particles employed are reported elsewhere [2]. The gelatinisation temperature range for starch was measured to be 64-69°C.

The Buoyancy Method for Manufacturing Syntactic Foams

The basic principles for manufacturing of syntactic foams containing starch as binder are based on the buoyancy of hollow microspheres in aqueous starch [2-3]. The starch binder (= starch particles + water) can be diluted for the purpose of controlling binder content in syntactic foam. Microspheres are dispersed in binder as a result of tumbling/stirring, thereafter it is left until the microspheres float to the surface, forming three phases: a top phase consisting of microspheres, starch particles and water, a middle phase of water only, and a bottom phase of microspheres, starch particles and water. The top phase is to be used for moulding. Gelatinisation of starch in the mixture was conducted after moulding, which is referred to as 'post-mould gelatinisation' as opposed to 'pre-mould gelatinisation' [2-3].

Phase Volume Measurement

Measuring cylinders (500 cc, 50 mm in diameter) were used for observation of phase separation and measurements of phase volumes at a room temperature ranging 17 - 20°C. A wooden lid was used on the top of each cylinder to minimise evaporation of water from the mixture. A constant volume of 400 cc for binder in each cylinder was used. Measurements for phase volumes were taken in every 30 minutes until a constant value was found in three successive readings and the final constant value was used. Measurements for phase volumes were made prior to adding microspheres. Starch particles (only two phases in this case, top phase contains water only and bottom phase contains starch particles and water) were found to settle down in 1 to 2 hours in binder, depending on starch content in binder. After adding microspheres to binder, phase separation took another 1 to 2 hours, depending on microsphere size and starch content in binder. Stirring of the mixture containing microspheres after sealing was achieved by manual end over end tumbling each measuring cylinder 20 times.

Results and Discussion

The phase volumes vary from initial bulk volumes after mixing microspheres and starch particles. The total volume change ratio after mixing in water (TVCRAM) is given as a function of starch volume fraction before mixing (SVFBM) ($= [\text{starch bulk volume in water before mixing}] / [\text{sum of microsphere and starch bulk volumes in water before mixing}]$) in Fig. 1. The TVCRAM is defined as the sum of top and bottom phase volumes after mixing divided by sum of microsphere and starch bulk volumes in water before mixing. It appears to be dependant upon microsphere size. As the microsphere mean size decreases, TVCRAM increases. This indicates that large gaps between microspheres and starch particles exist for small microspheres. Also, the maximum TVCRAM occurs at a starch volume fraction as indicated with filled and open arrows respectively for SL75 and SL300. (The position of the arrow will be further discussed below.) Further, the TVCRAM does not appear to be much affected by initial bulk volume of microspheres (IBVMS).

During the phase separation after tumbling of the aqueous mixture, the starch particles tend to settle to form a sediment while microspheres tend to float (due to their density differences). However, there is some interaction between microspheres and starch particles. Some starch particles are carried by microspheres to form the top phase, while some microspheres are carried by starch particles to form the bottom phase. To quantify this phase separation, bottom phase volume fraction after mixing (BPVFAM) ($= [\text{bottom phase volume after mixing}] / [\text{top and bottom phase volumes after mixing}]$) is plotted as a function of SVFBM, as shown in Fig. 2. If there were no

such interaction between microspheres and starch particles, all the data points would have been on the dashed line shown in Fig. 2. Data points under the dashed line indicate that starch particles are trapped in the top phase but those above the dashed line indicate that microspheres are trapped in the bottom phase. However, those that are close or on the dashed line do not necessarily mean that separation was complete. Thus, those data points close to the dashed line provide only a necessary condition (not sufficient condition) for the case where no interaction between microspheres and starch particles exists. Further, a physical transition (not graphical) in each size group is seen to occur at the similar starch volume fraction to that for TVCRAM (see Fig. 1) as indicated with filled and open arrows for SL75 and SL300 respectively.

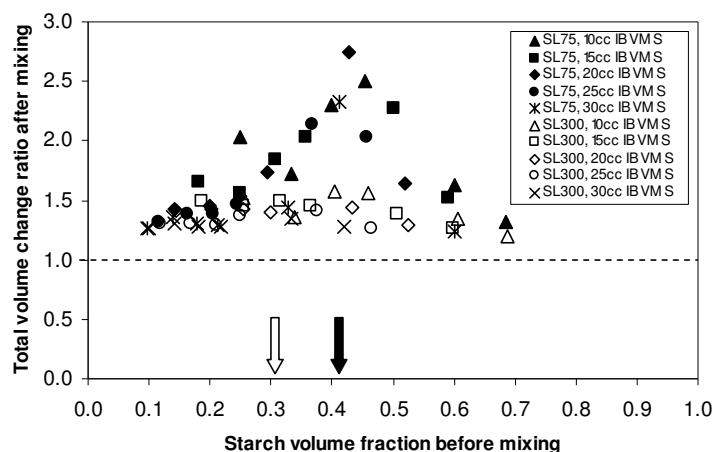


Figure 1 Total volume change ratio after mixing (TVCRAM) as a function of SVFBM.

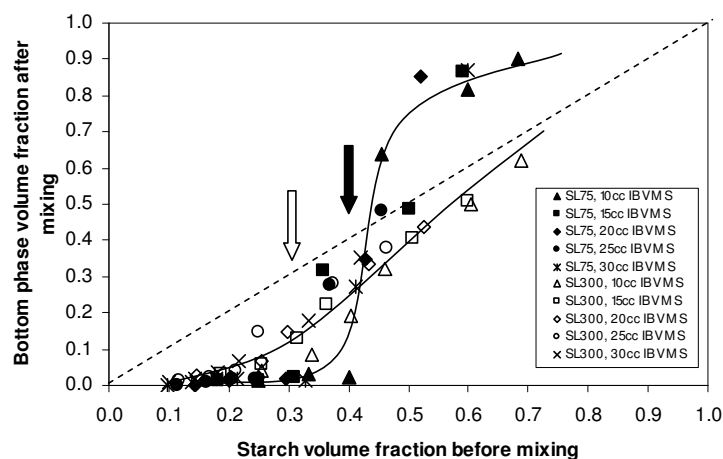


Figure 2 Bottom phase volume fraction after mixing (BPVFAM) as a function of SVFBM.

Direct microscopic observations indicated that the starch particles tend to adhere to microspheres. When a microsphere settles, starch particles that already adhered to the microsphere do not easily separate from the microsphere. However, starch particles settled on top of other starch particles already adhering to a microsphere readily separated when the microsphere motion changed from translational motion to rotational motion. This indicates that the attractive force between a starch particle and a microsphere is stronger than that between starch particles. Agglomerations are hence formed by starch particles acting as glue between microspheres. The buoyancy of each agglomerate depends upon composition – the more starch particles the heavier the agglomerate. The volume fraction of starch particles in an agglomerate consisting of one microsphere and multiple starch particles required to yield a relative density of 1.0 (VFSRD) was calculated to be 0.41 and 0.31 for SL75 and SL300 respectively. For these calculations, the mean

diameter of microspheres was used and it was assumed that starch particles are spherical. It is important to note that the values of VFSRD correspond to the points indicated by arrows in Fig. 1 and Fig. 2. Thus, the VFSRD appears a good indicator for both the maximum TVCRAM and the transitional points.

The use of the VFSRD as the transitional point indicator can be explained in statistical terms. Phase separation is likely to be a stochastic process. When the volume fraction of starch is lower than VFSRD, the density of an agglomeration is likely to be less than 1, allowing it to float to form the top phase. When a density of an agglomeration is higher than 1, it will settle to form the bottom phase. The abruptness of the transition (Fig. 2) for small microspheres can be explained as follows. It is a truism that smaller starch-microsphere inter-distance will allow more starch particle-microsphere collisions. As the microsphere size of a given microsphere bulk volume decreases in a given space, the number of microspheres will increase and starch-microsphere distances will decrease. As a result, agglomeration will occur more rapidly and will produce larger agglomerates. Consequently, relatively few individual particles or/and small agglomerations are formed when small microspheres are used. Such individual particles or/and small agglomerations are the ones that causes smoothness of the transition because their densities are not much affected and tend to follow the dashed line in Fig. 2.

Conclusions

The mixing process required for post-mould gelatinisation of manufacturing syntactic foams using starch as binder has been studied. A transition in the buoyancy of microsphere-starch phases was found at a particular volume fraction of starch. The transitional point was close to a volume fraction of starch at which a calculated relative density for a system consisting of multiple starch particles per microsphere was unity. Starch-microsphere inter-distance appears to be an important parameter affecting starch content in an agglomeration.

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